



Materials and Manufacturing

Introduction

Gail Chapline

Nondestructive Testing Innovations

Willard Castner

Patricia Howell

James Walker

Friction Stir Welding Advancements

Robert Ding

Jim Butler

Characterization of Materials in the Hydrogen Environment

Jon Frandsen

Jonathan Burkholder

Gregory Swanson

Space Environment: It's More Than a Vacuum

Lubert Leger

Steven Koontz

Chemical Fingerprinting

Michael Killpack

Environmental Assurance

Anne Meinhold

Unprecedented Accomplishments in the Use of Aluminum-Lithium Alloy

Preston McGill

Jim Butler

Myron Pessin

Orbiter Payload Bay Door

Lubert Leger

Ivan Spiker

To build a spacecraft, we must begin with materials. Sometimes the material choice is the solution. Other times, the design must accommodate the limitations of materials properties. The design of the Space Shuttle systems encountered many material challenges, such as weight savings, reusability, and operating in the space environment. NASA also faced manufacturing challenges, such as evolving federal regulations, the limited production of the systems, and maintaining flight certification. These constraints drove many innovative materials solutions. Innovations such as large composite payload bay doors, nondestructive materials evaluation, the super lightweight tank, and the understanding of hydrogen effects on materials were pathfinders used in today's industry. In addition, there were materials innovations in engineering testing, flight analysis, and manufacturing processes. In many areas, materials innovations overcame launch, landing, and low-Earth orbit operational challenges as well as environmental challenges, both in space and on Earth.



Nondestructive Testing Innovations

Have you ever selected a piece of fruit based on its appearance or squeezed it for that certain feel? Of course you have. We all have. In a sense, you performed a nondestructive test. Actually, we perform nondestructive testing every day. We visually examine or evaluate the things we use and buy to see whether they are suitable for their purpose. In most cases, we give the item just a cursory glance or squeeze; however, in some cases, we give it a conscious and detailed examination. We don't think of these routine examinations as nondestructive tests, but they are, and they give us a sense of what nondestructive testing is about.

Nondestructive testing is defined as the inspection or examination of materials, parts, and structures to determine their integrity and future usefulness without compromising or affecting their usefulness. The most fundamental nondestructive test of all is visual

inspection. In the industrial world, visual examination can be quite formal, with complex visual aids, pass/fail criteria, training requirements, and written procedures.

Nondestructive testing depends on incident or input energy that interacts with the material or part being examined. The incident or input energy can be modified by reflection from interaction within or transmission through the material or part. The process of detection and interpretation of the modified energy is how nondestructive testing provides knowledge about the material or part. Tests range from the simple detection and interpretation of reflected visible light by the human eye (visual examination) to the complex electronic detection and mathematical reconstruction of through-transmitted x-radiation (computerized axial tomography [CAT] scan). From a nondestructive testing perspective, the similarity between the simple visual examination and the complex CAT scan is the input energy (visible light vs. x-rays) and the modified energy

(detected by the human eye vs. an electronic x-ray detector).

Nondestructive testing is a routine part of a spacecraft's life cycle. For the reusable shuttle, nondestructive testing began during the manufacturing and test phases and was applied throughout its service life. NASA performed many such nondestructive tests on the shuttle vehicles and developed most nondestructive testing innovations in response to shuttle problems.

Quantitative Nondestructive Testing of Fatigue Cracks

One of the most significant nondestructive testing innovations was quantifying the flaw sizes that conventional nondestructive testing methods could reliably detect. NASA used artificially induced fatigue cracks to make the determination because such flaws were relatively easy to grow and control, hard to detect, and tended to bound the population of flaws of interest. The need to quantify the reliably detectable crack sizes was



Two examples of the most basic nondestructive testing:

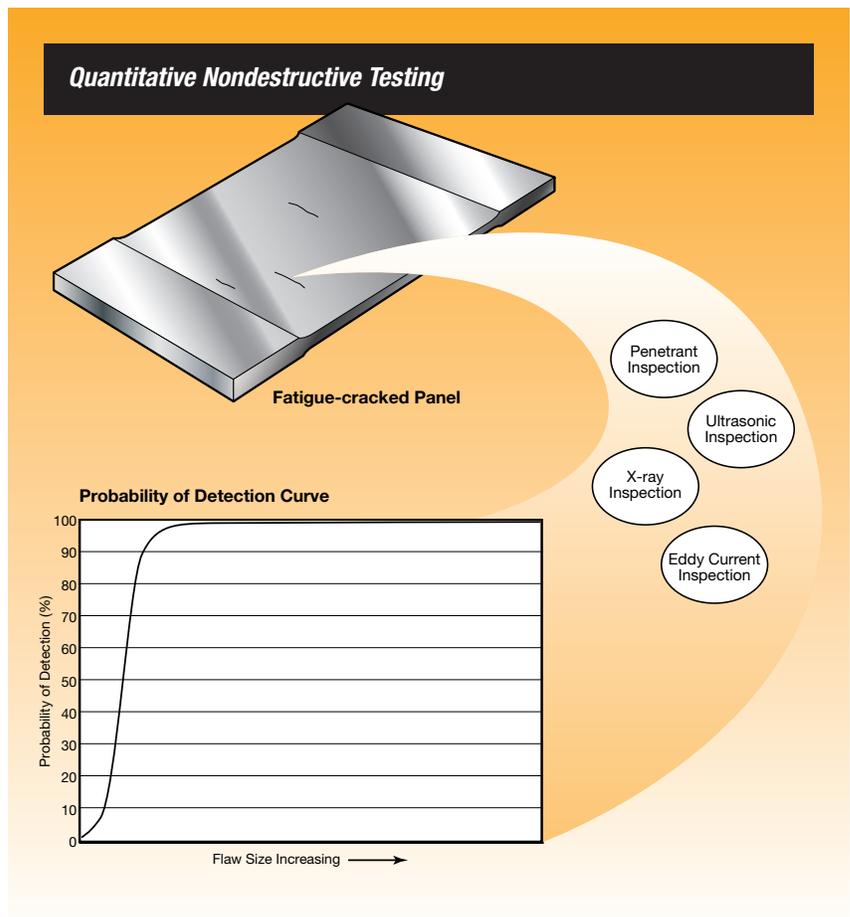
Left, a gardener checks ripening vegetables. Right, Astronaut Eileen Collins, STS-114 (2005) mission commander, looks closely at a reinforced carbon-carbon panel on one of the wings of the Space Shuttle Atlantis in the Orbiter Processing Facility at Kennedy Space Center (KSC). Collins and the other crew members were at KSC to take part in hands-on equipment and Orbiter familiarization.



mandated by a fracture control interest in having confidence in the starting crack size that could be used in fracture and life calculations. Although there was no innovation of any specific nondestructive testing method, quantifying—in a statistical way—the reliably detectable crack sizes associated with the conventional nondestructive evaluation methods was innovative and led the way to the adoption of similar quantitative nondestructive evaluation practices in other industries.

The quantification of nondestructive testing methods is commonly referred to today as probability of detection. The Space Shuttle Program developed some of the earliest data for the penetrant, x-ray, ultrasonic, and eddy current nondestructive testing methods—the principal nondestructive testing methods used to inspect shuttle components during manufacturing. Data showed that inspectors certified to aerospace inspection standards could, on average, perform to a certain probability of detection level defined as standard nondestructive evaluation.

Beyond standard nondestructive evaluation, NASA introduced a special nondestructive evaluation level of probability of detection wherein the detection of cracks smaller than the standard sizes had to be demonstrated by test. Engineers fabricated fatigue-cracked specimens that were used over many years to certify and recertify, by test, the inspectors and their nondestructive evaluation processes to the smaller, special nondestructive evaluation crack size. The size of the fatigue cracks in the specimens was targeted to be a surface-breaking semicircular crack 0.127 cm (0.050 in.) long by 0.063 cm (0.025 in.) deep, a size that was significantly smaller than the standard nondestructive evaluation crack size of 0.381 cm (0.150 in.) long by 0.19 cm (0.075 in.) deep.



The special probability of detection specimen sets typically consisted of 29 randomly distributed cracks of approximately the same size. By detecting all 29 cracks, the inspector and the specific nondestructive evaluation process were considered capable of detecting the crack size to a 90% probability of detection with 95% confidence.

Nondestructive Testing of Thermal Protection System Tiles

The development of Thermal Protection System tiles was one of the most unique and difficult developments of the program. Because of this material's "unknowns," the tile attachment scheme, and their extremely fragile

nature, NASA examined a number of nondestructive testing methods.

Acoustic Emission Monitoring

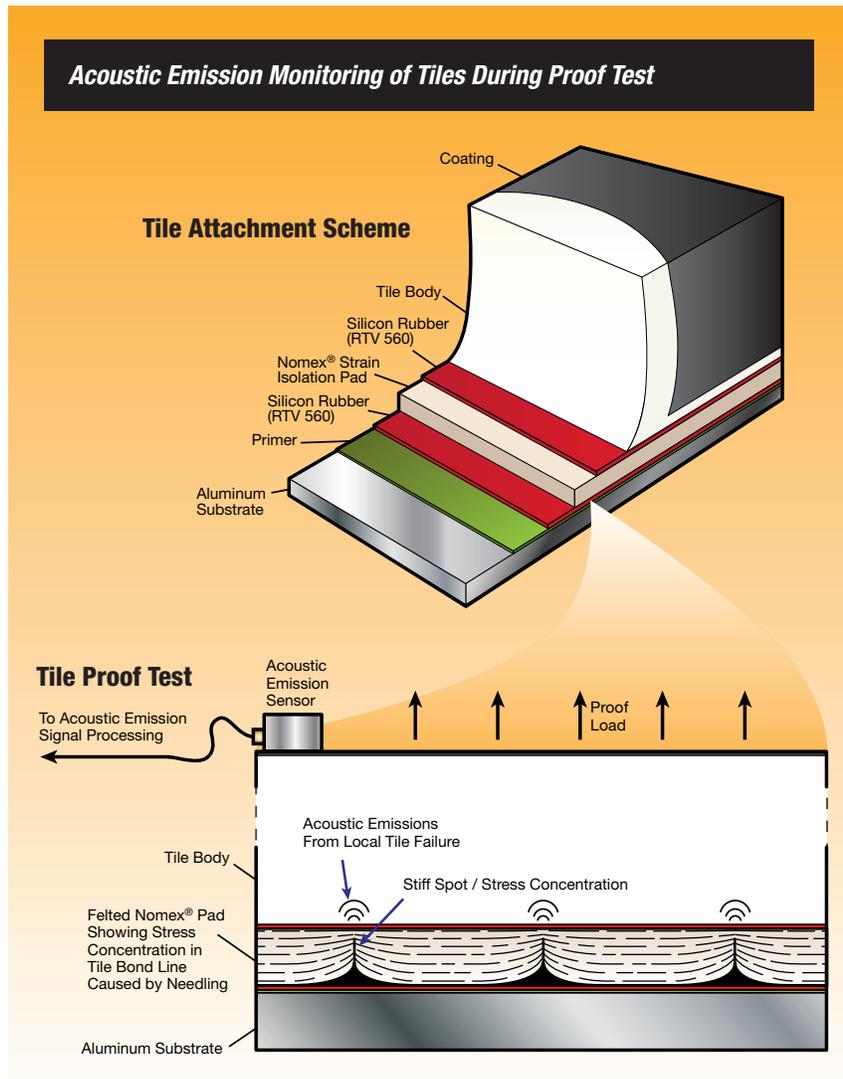
Late in the development of the shuttle Thermal Protection System and just before the first shuttle launch, NASA encountered a major problem with the attachment of the tiles to the Orbiter's exterior skin. The bond strength of the tile system was lower than the already-low strength of the tile material, and this was not accounted for in the design. The low bond strength was due to stress concentrations at the tile-to-strain isolation pad bond line interface. A Nomex® felt strain isolation pad was bonded between each tile and the Orbiter skin to minimize the



lateral strain input to the tile from the aluminum skin. These stress concentrations led to early and progressive failures of the tile material at the tile-to-strain isolation pad bond line interface when the tile was loaded.

To determine whether low bond strengths existed, engineers resorted to proof testing for each tile. This required thousands of individual tile proof tests prior to first flight. Space Shuttle Columbia (Space Transportation System [STS]-1) was at Kennedy Space Center being readied for first flight when NASA decided that proof testing was necessary. Since proof testing was not necessarily nondestructive and tiles could be damaged by the test, NASA sought a means of monitoring potential damage; acoustic emission nondestructive testing was an obvious choice. The acoustic signatures of a low bond strength tile or a tile damaged during proof test were determined through laboratory proof testing of full-size tile arrays.

To say that the development and implementation of acoustic emission monitoring during tile proof testing was done on a crash basis would be an understatement. The fast pace was dictated by a program that was already behind schedule, and the tile bond strength problem threatened significant additional delay. At the height of the effort, 18 acoustic emission systems with fully trained three-person crews were in operation 24 hours a day, 7 days a week. The effort was the largest single concentration of acoustic emission equipment at a single job site. As often happens with such problems, where one solution can be overtaken and replaced by another, a tile densification design fix for the low-strength bond was found and implemented prior to first flight, thus obviating the need for continued



acoustic emission monitoring. By the time the acoustic emission monitoring was phased out, NASA had performed 20,000 acoustic emission monitored proof tests.

Sonic Velocity Testing

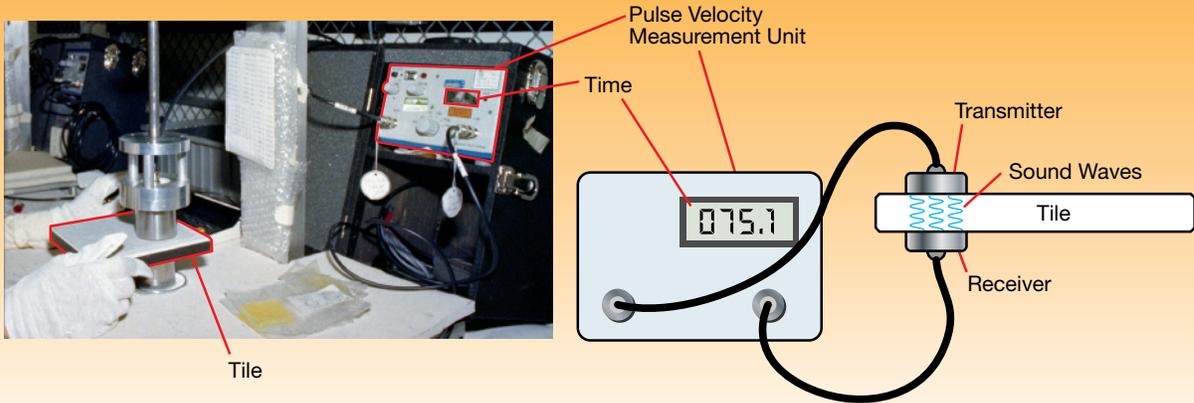
Another early shuttle nondestructive testing innovation was the use of an ultrasonic test technique to ensure that the Thermal Protection System tiles were structurally sound prior to installation. Evaluation of pulse or sonic velocity tests showed a velocity relationship with respect to

both tile density and strength. These measurements could be used as a quality-control tool to screen tiles for low density and low strength and could also determine the orientation of the tile.

The sonic velocity technique input a short-duration mechanical impulse into the tile. A transmitting transducer and a receiving transducer, placed on opposite sides of the tile, measured the pulse's transit time through the tile. For the Lockheed-provided tile material, LI-900 (with bulk density of 144 kg/m³ [9 pounds/ft³]), the average through-the-thickness sonic velocity was on the



Sonic Velocity Testing of Tiles at Kennedy Space Center Thermal Protection System Facility
The speed of sound through the tile is related to density and strength.



order of 640 m/sec (2,100 ft/sec), and the through-the-thickness flat-wise tensile strength was on the order of 1.69 kg/cm² (24 pounds/in²). The LI-900 acceptance criterion for sonic velocity was set at 518 m/sec (1,700 ft/sec), which corresponded to a minimum strength of 0.91 kg/cm² (13 pounds/in²). Sonic velocity testing was phased out in the early 1990s.

Post-Columbia Accident Nondestructive Testing of External Tank

A consequence of the Columbia (STS-107) accident in 2003 was the development of several nondestructive innovations, including terahertz imaging and backscatter radiography of External Tank foam and thermography of the reinforced carbon-carbon—both on orbit and on the ground—during vehicle turnaround. The loss of foam, reinforced carbon-carbon impact damage, and on-orbit inspection of Thermal Protection System damage were all problems that could be mitigated to some extent through the application of nondestructive testing methods.

Nondestructive Testing of External Tank Spray-on Foam Insulation

Prior to the Columbia accident, no nondestructive testing methods were available for External Tank foam inspection, although NASA pursued development efforts from the early 1980s until the early 1990s. The foam was effectively a collection of small air-filled bubbles with thin polyurethane membranes, making the foam a thermal and electrical insulator with very high acoustic attenuation. Due to these properties, it was not feasible to inspect the foam with conventional methods such as eddy current, ultrasonics, or thermography. In addition, since the foam was considered nonstructural, problems of delaminations occurring during foam application and foam popping off (“popcorning”) during ascent were considered manageable through process control.

After the Columbia accident, NASA focused on developing nondestructive testing methods for finding voids and delaminations in the thick, hand-sprayed foam applications around protuberances and closeout

areas. The loss of foam applied to the large areas of the tank was not as much of concern because the automated acreage spray-on process was better controlled, making it more unlikely to come off. In the event it did come off, the pieces would likely be small because acreage foam was relatively thin. NASA’s intense focus resulted in the development and implementation of two methods for foam inspection—terahertz imaging and backscatter radiography—that represented new and unique application of nondestructive inspection methods.

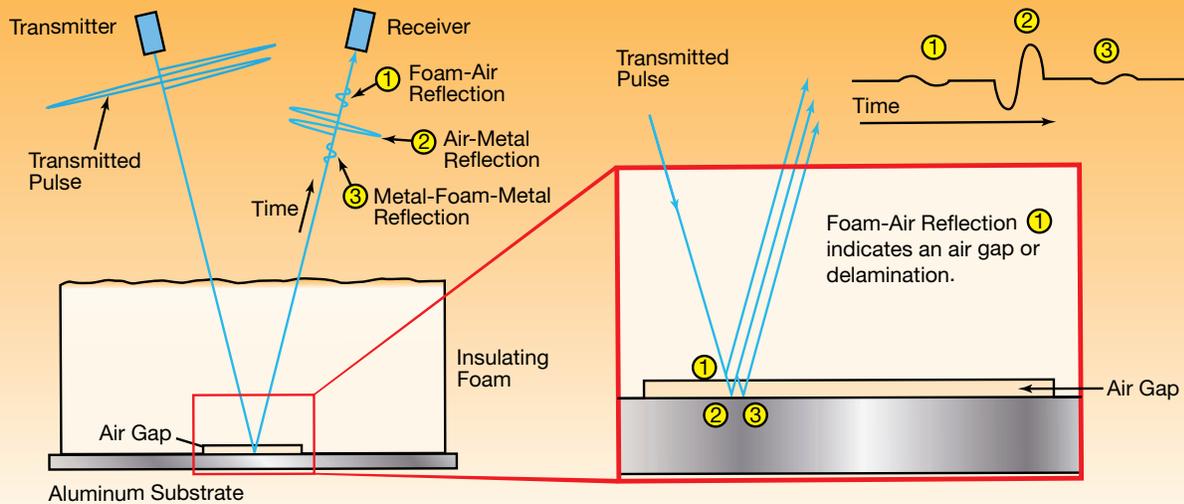
Terahertz Imaging

Terahertz imaging is a method that operates in the terahertz region of the electromagnetic spectrum between microwave frequencies and far-infrared frequencies. Low-density hydrocarbon materials like External Tank foam were relatively transparent to terahertz radiation. Terahertz imaging used a pulser to transmit energy into a structure and a receiver to record the energy reflected off the substrate or internal defects. As the signal traveled through the structure, its basic wave



Terahertz Imaging System

This system uses high-frequency electromagnetic pulses.



properties were altered by the attenuation of the material and any internal defects. An image was made by scanning the pulser/receiver combination over the foam surface and displaying the received signal.

Probability of detection studies of inserted artificial voids showed around 90% detection of the larger voids in simple geometries, but less than 90% detection in the more-complicated geometries of voids around protrusions. Further refinements showed that delaminations were particularly difficult to detect. The detection threshold for a 2.54-cm- (1-in.-)diameter laminar defect was found to be a height of 0.508 cm (0.2 in.), essentially meaning delaminations could not be detected. The terahertz inspection method was used for engineering evaluation, and any defects found were dealt with by an engineering review process.

Backscatter Radiography

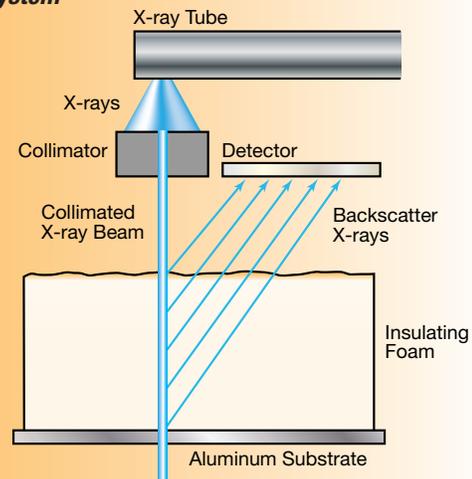
Backscatter radiography uses a conventional industrial x-ray tube to generate a collimated beam of x-rays

that is scanned over the test object. The backscattering of x-rays results from the Compton effect—or scattering—in which absorption of the incident or primary x-rays by the atoms of the

Backscatter X-ray Imaging System



Insulating foam covers the External Tank.



An irradiated column of foam that has voids produces less backscattered x-rays than a void-free column of foam.



test material are reradiated at a lower energy as secondary x-rays in all directions. The reradiated or backscattered x-rays were collected in collimated radiation detectors mounted around the x-ray source. Voids or defects in the test material were imaged in backscatter radiography in the same manner as they were in conventional through-transmission radiography. Imaging of voids or defects depended on less absorbing material and less backscattered x-rays from the void.

Since only the backscattered x-rays were collected, the technique was single sided and suited for foam inspection. The foam was well suited for backscatter radiography since Compton scattering is greater from low atomic number materials. The technique was more sensitive to near surface voids but was unable to detect delaminations. Like terahertz imaging, backscatter radiography was used for engineering evaluation, and defects found were dealt with by an engineering review process.

Nondestructive Testing of Reinforced Carbon-Carbon System Components

A recommendation of the Columbia Accident Investigation Board stated: “Develop and implement a comprehensive inspection plan to determine the structural integrity of all Reinforced Carbon-Carbon (RCC) system components. This inspection plan should take advantage of advanced non-destructive inspection technology.” To comply with this recommendation, NASA investigated advanced inspection technology for inspection of the reinforced carbon-carbon leading edge panels during ground turnarounds and while on orbit.

Ground Turnaround Thermography

NASA selected infrared flash thermography as the method to determine the structural integrity of the reinforced carbon-carbon components. Thermography was a fast, noncontacting, one-sided application that was easy to implement in the Orbiter’s servicing environment.



Infrared thermography inspection of the Orbiter nose captured at the instant of the xenon lamp flash. Kennedy Space Center Orbiter Processing Facility.

The Thermographic Inspection System was an active infrared flash thermography system. Thermographic inspection examined and recorded the surface temperature transients of the test article after application of a short-duration heat pulse. The rate of heat transfer away from the test article surface depended on the thermal diffusivity of the material and the uniformity and integrity of the test material. Defects in the material would retard the heat flow away from the surface, thus producing surface temperature differentials that were reflective of the uniformity of the material and its defect content. A defect-free material would uniformly

transfer heat into the underlying material, and the surface temperature would appear the same over the entire test surface; however, a delamination would prevent or significantly retard heat flow across the gap created by the delamination, resulting in more-local heat retention and higher surface temperature in comparison to the material surrounding the delamination. Temperature differences were detected by the infrared camera, which provided visual images of the defects. Electronic signals were processed and enhanced for easier interpretation. The heat pulse was provided by flashing xenon lamps in a hooded arrangement that excluded ambient light. The infrared camera was transported along a floor-mounted rail system in the Orbiter Processing Facility for the leading edge panel inspections, allowing full and secure access to all of the leading edge surfaces. After the transport cart was positioned, the camera was positioned manually via a grid system that allowed the same areas to be compared from flight to flight.

The thermography system was validated on specimens containing flat bottom holes of different diameters and depths. Validation testing confirmed the ability of the flash thermography system to detect the size holes that needed to be detected.

After the first Return to Flight mission—STS-114 (2005)—the postflight thermography inspection discovered a suspicious indication in the joggle area of a panel. Subsequent investigation showed that the indication was a delamination. This discovery set in motion an intense focus on joggle-area delaminations and their characterization and consequence. Many months of further tests, development, and refinement of the thermography methodology



determined that critical delaminations would be detected and sized by flash thermography and provided the basis for flightworthiness.

On-orbit Thermography

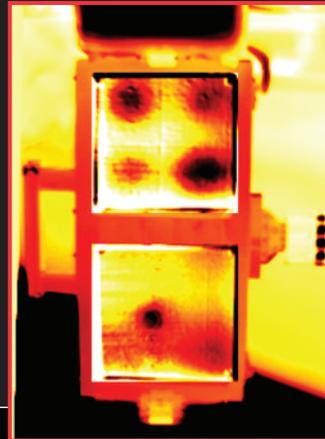
The success of infrared thermography for ground-based turnaround inspection of the wing leading edge panels and the extensive use of thermography during Return to Flight impact testing made it the choice for on-orbit inspection of the leading edge reinforced carbon-carbon material. A thermal gradient through the material must exist to detect subsurface reinforced carbon-carbon damage with infrared thermography. A series of ground tests demonstrated that sunlight or solar heating and shadowing could be used to generate the necessary thermal gradient, which significantly simplified the camera development task.

With the feasibility of on-orbit thermography demonstrated and with the spaceflight limitations on weight and power taken into account, NASA selected a commercial off-the-shelf microbolometer camera for modification and development into a space-qualified infrared camera for inspecting the reinforced carbon-carbon for impact damage while on orbit.

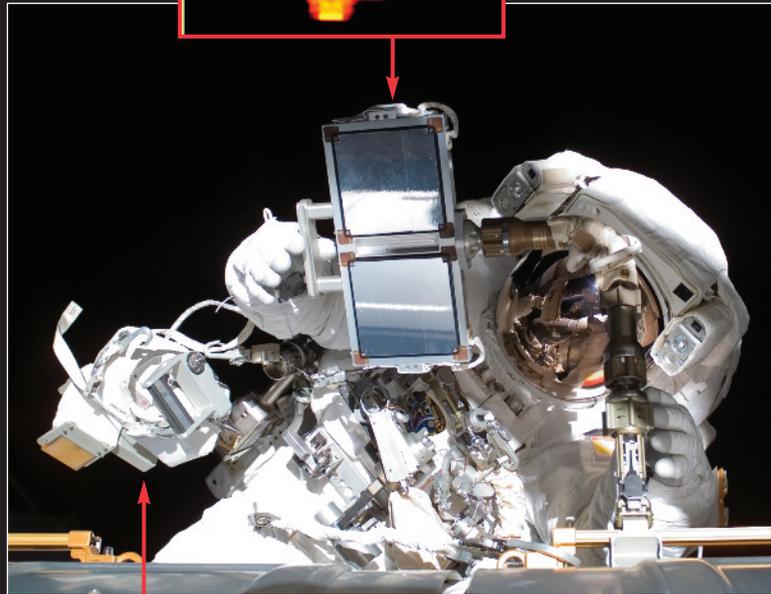
The extravehicular activity infrared camera operated successfully on its three flights. Two reinforced carbon-carbon test panels with simulated damage were flown and inspected on STS-121 (2006). The intentional impact damage in one panel and the flat bottom holes in the other panel were clearly imaged. Engineers also performed a similar on-orbit test on two other intentionally damaged reinforced carbon-carbon test panels during a space station extravehicular activity with the

On-orbit Thermography

Processed infrared images of reinforced carbon-carbon test panels.



Astronaut Thomas Reiter mounting pre-damaged reinforced carbon-carbon test panels on the International Space Station during STS-121 (2006).



Extravehicular activity infrared flight camera.



same result of clearly imaging the damage. The end result of these efforts was a mature nondestructive inspection technique that was transitioned and demonstrated as an on-orbit nondestructive inspection technique.

Additional Nondestructive Testing

Most nondestructive testing innovations resulted from problems that the shuttle encountered over the years, where nondestructive testing provided all or part of the solution. Other solutions worth mentioning include: ultrasonic extensometer measurements of critical shuttle bolt tensioning; terahertz imaging of corrosion under tiles; phased array ultrasonic testing of the External Tank friction stir welds and the shuttle crawler-transporter shoes; thermographic leak detection of the main engine nozzle; digital radiography of Columbia debris; surface replication of flow liner cracks; and the on-board wing leading edge health monitoring impact system.

Friction Stir Welding Advancements

NASA invents welding fixture.



Friction stir welding units, featuring auto-adjustable pin tools, welded External Tank barrel sections at NASA's Michoud Assembly Facility in New Orleans, Louisiana. The units measured 8.4 m (27.5 ft) in diameter and approximately 7.6 m (25 ft) tall to accommodate the largest barrel sections.

In the mid 1990s, NASA pursued the implementation of friction stir welding technology—a process developed by The Welding Institute of Cambridge, England—to improve External Tank welds. This effort led to the invention of an auto-adjustable welding pin tool adopted by the Space Shuttle Program, the Ares Program (NASA-developed heavy launch vehicles), and industry.

Standard fusion-welding techniques rely on torch-generated heat to melt and join the metal. Friction stir welding does not melt the metal. Instead, it uses a rotating pin and “shoulder” to generate friction, stir the metal together, and forge a bond. This process results in welds with mechanical properties superior to fusion welds.

Standard friction stir welding technology has drawbacks, however; namely, a non-adjustable pin tool that leaves a “keyhole” at the end of a circular weld and the inability to automatically adjust the pin length for materials of varying thickness. NASA's implementation of friction stir welding for the External Tank resulted in the invention and patenting of an auto-adjustable pin tool that automatically retracts and extends in and out of the shoulder. This feature provides the capability to make 360-degree welds without leaving a keyhole, and to weld varying thicknesses.

During 2002-2003, NASA and the External Tank prime contractor, Lockheed Martin, implemented auto-adjustable pin tool friction stir welding for liquid hydrogen and liquid oxygen tank longitudinal welds. Since that time, these friction stir welds have been virtually defect-free. NASA's invention was being used to weld Ares upper-stage cryogenic hardware. It has also been adopted by industry and is being used in the manufacturing of aerospace and aircraft frames.



Characterization of Materials in the Hydrogen Environment

From the humid, corrosion-friendly atmosphere of Kennedy Space Center, to the extreme heat of ascent, to the cold vacuum of space, the Space Shuttle faced one hostile environment after another. One of those harsh environments—the hydrogen environment—existed within the shuttle itself. Liquid hydrogen was the fuel that powered the shuttle's complex, powerful, and reusable main engine. Hydrogen provided the high specific impulse—the bang per pound of fuel needed to perform the shuttle's heavy-lifting duties. Hydrogen, however, was also a potential threat to the very metal of the propulsion system that used it.

The diffusion of hydrogen atoms into a metal can make it more brittle and prone to cracking—a process called hydrogen embrittlement. This effect can reduce the toughness of carefully selected and prepared materials. A concern that exposure to hydrogen might encourage crack growth was present from the beginning of the Space Shuttle Program, but the rationale for using hydrogen was compelling.

The Challenge of the Hydrogen Environment

Hydrogen embrittlement posed more than a single engineering problem for the Space Shuttle. This was partly because hydrogen embrittlement can occur in three different ways. The most common mode occurs when hydrogen is absorbed by a material that is relatively unstressed, such as the components of the shuttle's main

engines before they experienced the extreme loads of liftoff and flight; this is called internal hydrogen embrittlement. Under the right conditions, internal hydrogen embrittlement has the potential to render materials too weak and brittle to survive high stresses applied later.

Alternatively, embrittlement can affect a material that is immersed in hydrogen while the material is being stressed and deformed. This phenomenon is called hydrogen environment embrittlement, which can occur in pressurized hydrogen storage vessels. These vessels are constantly stressed while in contact with hydrogen. Hydrogen environment embrittlement can potentially reduce ductility over time and enable cracking, or hydrogen may simply reduce the strength of a vessel until it is too weak to bear its own pressure.

Finally, hydrogen can react chemically with elements that are present in a metal, forming inclusions that can degrade the properties of that metal or even cause blisters on the metal's surface. This effect is called hydrogen reaction embrittlement. In the shuttle's main engine components, the reaction between hydrogen and the titanium alloys occurred to internally form brittle titanium hydrides, which was most likely to occur at locations where there were high tensile stresses in the part. Hydrogen reaction embrittlement can affect steels when hydrogen atoms combine with the carbon atoms dissolved in the metal. Hydrogen reaction embrittlement can also blister copper when hydrogen reacts with the internal oxygen in a solid copper piece, thereby forming steam blisters.

Insights on Hydrogen Environment Embrittlement

NASA studied the effects of hydrogen embrittlement in the 1960s. In the early 1970s, the scope of NASA-sponsored research broadened to include hydrogen environment embrittlement effects on fracture and fatigue. Engineers immersed specimens in hydrogen and performed a battery of tests. They applied repeated load cycles to specimens until they fatigued and broke apart; measured crack growth rates in cyclic loading and under a constant static load; and tested materials in high-heat and high-pressure hydrogen environments. Always, results were compared for each material to its performance in room-temperature air.

During the early years of the Space Shuttle Program, NASA and contractor engineers made a number of key discoveries regarding hydrogen environment embrittlement. First, cracks were shown to grow faster when loaded in a hydrogen environment. This finding would have significant implications for the shuttle design, as fracture assessments of the propulsion system would have to account for accelerated cracking. Second, scientists observed that hydrogen environment embrittlement could result in crack growth under a constant static load. This behavior was unusual for metals. Ductile materials such as metals tend to crack in alternating stress fields, not in fixed ones, unless a chemical or an environmental cause is present. Again, the design of the shuttle would have to account for this effect. Finally, hydrogen environment embrittlement was shown to have more severe effects at higher pressures. Intriguingly, degradation of tensile properties was found to be proportional to the square root of pressure.



The overall approach to hydrogen environment embrittlement research was straightforward. As a matter of common practice, NASA characterized the strength and fracture behavior of its alloys. To determine how these alloys would tolerate hydrogen, engineers simply adapted their tests to include a high-pressure hydrogen environment. After learning that high pressure exacerbates hydrogen environment embrittlement, they further adapted the tests to include a hydrogen pressure of 703 kg/cm² (10,000 psi). Later in the program, materials being considered for use in the main engine were tested at a reduced pressure of 492 kg/cm (7,000 psi) to be more consistent with operation conditions. The difference between room-temperature air material property data and these new results was a measurable effect of hydrogen environment embrittlement. Now that these effects could be quantified, the next step was to safeguard the shuttle.

Making Parts Resistant to Hydrogen Environment Embrittlement

One way to protect the main engines from hydrogen environment embrittlement was through materials selection. NASA chose naturally resistant materials when possible. There were, however, often a multitude of conflicting demands on these materials: they had to be lightweight, strong, tough, well suited for the manufacturing processes that shaped them, weldable, and able to bear significant temperature swings. The additional constraint of imperviousness to hydrogen environment embrittlement was not always realistic, so engineers

experimented with coatings and plating processes. The concept was to shield vulnerable metal from any contact with hydrogen. A thin layer of hydrogen environment embrittlement-resistant metal would form a barrier that separated at-risk material from hydrogen fuel.

Engineers concentrated their research on coatings that had low solubility and low-diffusion rates for hydrogen at room temperature. Testing had demonstrated that hydrogen environment embrittlement is worst at near-room temperature, so NASA selected coatings based on their effectiveness in that range. The most efficient barrier to hydrogen, engineers found, was gold plating; however, the cost of developing gold plating processes was a significant factor. Engineers observed that copper plating provided as much protection as gold, as long as a thicker and heavier layer was applied.

Protecting weld surfaces was often more challenging. The weld surfaces exposed to hydrogen fuel during flight were typically not accessible to plating after the weld was complete. Overcoming this problem required a more time-consuming and costly approach. Engineers developed weld overlays, processes in which hydrogen environment embrittlement-resistant filler metals were added during a final welding pass. These protective fillers sealed over the weld joints and provided the necessary barrier from hydrogen. NASA used overlays in combination with plating of accessible regions to prevent hydrogen environment embrittlement in engine welds.

These approaches—a combination of two or more hydrogen environment embrittlement prevention methods—were the practical solution for many of the embrittlement-vulnerable parts of the engines. For example, the most heavily used alloy in the engines was Inconel® 718, an alloy known to be affected by hydrogen environment embrittlement. Engineers identified an alternative heat treatment, different from the one typically used, which limited embrittlement. But this alone was insufficient. In the most critical locations, the alternative heat treatment was combined with copper plating and weld overlays.

A unique processing approach was also used to prevent embrittlement in the engine's main combustion chamber. This chamber was made with a highly conductive copper alloy. Its walls contained cooling channels that circulated cold liquid hydrogen and kept the chamber from melting in the extreme heat of combustion. But the hydrogen-filled channels became prone to hydrogen environment embrittlement. These liquid hydrogen channels were made by machining slots in the copper and then plated with nickel, which closed out the open slot and formed a coolant channel. The nickel plate cracked in the hydrogen environment and reduced the pressure capability of the channels. Engineers devised a two-part solution. First, they developed an alternative heat treatment to optimize nickel's performance in hydrogen. Next, they coated the nickel with a layer of copper to isolate it from the liquid hydrogen. This two-pronged strategy worked, and liquid hydrogen could be safely used as the combustion chamber coolant.



Addressing Internal Hydrogen Embrittlement

Whereas hydrogen environment embrittlement was of great concern at NASA in the 1960s, internal hydrogen embrittlement was largely dismissed even through the early years of the Space Shuttle Program. Internal hydrogen embrittlement had never been a significant problem for the types of materials used in spaceflight hardware. The superalloys and particular stainless steels selected by NASA were thought to be resistant to internal hydrogen embrittlement. Engineers thought the face-centered, cubic, close-packed crystal structure would leave too little room for hydrogen to permeate and diffuse.

Recall that internal hydrogen embrittlement occurs when hydrogen is absorbed before high operational stresses. Hydrogen enters into the metal and remains there, making it more brittle and likely to crack when extreme service loads are applied later. It is the accumulation of absorbed hydrogen, rather than the immediate exposure at the moment of high stress, that compromises an internal hydrogen embrittlement-affected material. When NASA initially designed the main engine, engineers accounted for hydrogen absorbed during manufacturing. Engineers, however, thought that the materials that were formed and processed without collecting a significant amount of hydrogen were not in danger of absorbing considerable amounts later.

This notion about internal hydrogen embrittlement was challenged during the preparation of an engine failure analysis document in 1988. The engine

was repeatedly exposed to hydrogen in flight and after flight, at high temperatures and extreme pressure. The report suggested that in these exceptional heat and pressure conditions some engine materials might, in fact, gather small amounts of hydrogen with each flight. Gradually, over time, these materials could accumulate enough hydrogen to undermine ductility.

Engineers developed a special test regimen to screen materials for high-temperature, high-pressure hydrogen accumulation. Test specimens were “charged” with hydrogen at 649°C (1,200°F) and 351.6 kg/cm² (5,000 psi). They were then quickly cooled and tested for strength and ductility under normal conditions. Surprisingly, embrittlement by internal hydrogen embrittlement was observed to be as severe as by hydrogen environment embrittlement. As a subsequent string of fatigue tests confirmed this comparison, NASA had to reevaluate its approach to preventing hydrogen embrittlement. The agency’s focus on hydrogen environment embrittlement had been a near-total focus. Now, a new awareness of internal hydrogen embrittlement would drive a reexamination.

Fortunately, the process for calculating design properties from test data had been conservative. The margins of safety were wide enough to bound the combined effects of internal hydrogen embrittlement and hydrogen environment embrittlement. The wealth of experience gained in studying hydrogen environment embrittlement and mitigating its effects also worked in NASA’s favor. Some of the same methodologies could now be applied to

internal hydrogen embrittlement. For instance, protective plating would operate on the same principle—the creation of a barrier between hydrogen and a vulnerable alloy—whether hydrogen environment embrittlement or internal hydrogen embrittlement was the chief worry. Continued testing of “charged” specimens would allow quantification of internal hydrogen embrittlement damage, just as hydrogen immersion testing had enabled measurement of hydrogen environment embrittlement effects.

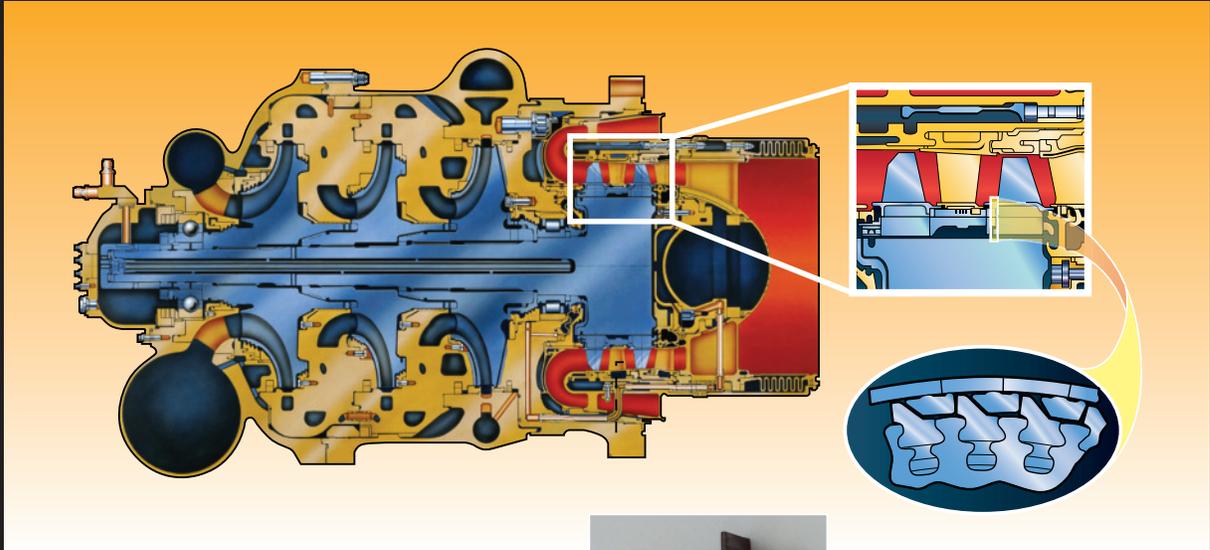
Taking strategies generated to avoid hydrogen environment embrittlement and refitting them to prevent internal hydrogen embrittlement, however, often required additional analysis. For example, from the beginning of the Space Shuttle Program NASA used coatings to separate at-risk metals from hydrogen. The agency intentionally chose these coatings for their performance at near-room temperature, when hydrogen environment embrittlement is most aggressive. Tests showed the coatings were less effective in the high heat that promotes internal hydrogen embrittlement. New research and experimentation was required to prove that these protective coatings were adequate—that, although they didn’t completely prevent the absorption of hydrogen when temperatures and pressures were extreme, they did reduce it to safe levels.

Special Cases: High-Pressure Fuel Turbopump Housing

NASA encountered a unique hydrogen embrittlement issue during development testing of the main engine high-pressure fuel turbopump.



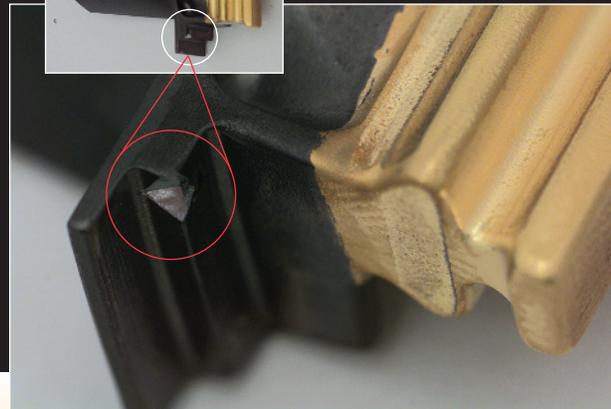
High-Pressure Fuel and Oxidizer Turbopump Turbine Blade Cracks



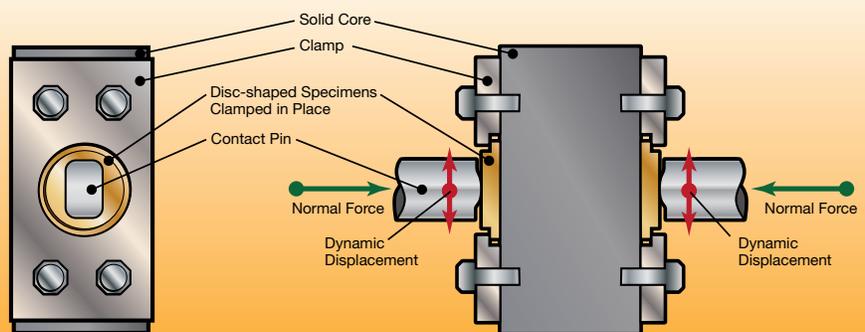
After observing cracks on polycrystalline turbine blades, NASA redesigned the blades as single-crystal parts. When tested in hydrogen, cracks were detected. Scientists used a Brazilian disc test to create the tensile and shear stresses that had caused growth. NASA resolved cracking in the airfoil with changes that eliminated stress concentrations and smoothed the flow of molten metal during casting. To assess cracking at damper contacts, scientists extracted test specimens from single crystal bars, machined contact pins from the damper material, and loaded two specimens. This contact fixture was supported in a test rig that allowed the temperature, loads, and load cycle rate to be varied. Specimens were pre-charged with hydrogen, tested at elevated temperatures, and cycled at high frequency to actual operating conditions.



First Stage Blade 42 Trailing Edge Root



Schematic of Test Rig





A leak developed during the test; this leak was traced to cracks in the mounting flange of the turbopump's housing. The housing was made from embrittlement-prone nickel-chromium alloy Inconel® 718, and the cracks were found to originate in small regions of highly concentrated stress. So, engineers changed the material to a more-hydrogen-tolerant alloy, Inconel® 100, and they redesigned the housing to reduce stress concentrations. This initially appeared to solve the problem. Then, cracks were discovered in other parts of the housing. Structural and thermal analysis could not explain this cracking. The locations and size of the cracks did not fit with existing fatigue and crack-growth data.

To resolve this inconsistency, engineers considered the service conditions of the housing. The operating environment of the cracked regions was a mixture of high-pressure hydrogen and steam at 149°C to 260°C (300°F to 500°F). Generally, hydrogen environment embrittlement occurs near room temperature and would not be a significant concern at that level of heat; however, because of the unexplained cracking, a decision was made to test Inconel® 100 at elevated temperatures in hydrogen and hydrogen mixed with steam. Again, the results were unexpected. Engineers observed a pronounced reduction in strength and ductility in these environments at elevated temperatures. Crack growth occurred at highly accelerated rates—as high as two orders of magnitude above room-temperature air when the crack was heavily loaded to 30 ksi $\sqrt{\text{in}}$ (33 MPa $\sqrt{\text{m}}$) and held for normal engine operating time. Moreover, crack growth was driven by both the number of load cycles and the duration of each load cycle. Crack growth is typically sensitive to the number and magnitude of load cycles but not to the length of time for each cycle.

Clearly, the combination of the hydrogen and steam mixture and the uncommonly high stress concentrations was promoting hydrogen environment embrittlement in Inconel® 100 at high temperatures. Resolving this issue required three modifications. First, detailed changes to the shape of the housing were made, further reducing stress concentrations. Second, gold plating was added to shield the Inconel® 100 from the hot hydrogen and steam mixture. Finally, a manufacturing process called “shot peening” was used to fortify the surface of the housing against tensile stresses by impacting it with shot, determined to be promoting fracture, and therefore eliminated.

Summary

The material characterization done in the design phase of the main engine, and the subsequent anomaly resolution during its development phase, expanded both the material properties database and the understanding of hydrogen embrittlement. The range of hydrogen embrittlement data has been broadened from essentially encompassing only steels to now including superalloys. It was also extended from including primarily tensile properties to including extensive low-cycle fatigue and fracture-mechanics testing in conditions favorable to internal hydrogen embrittlement or hydrogen environment embrittlement. The resultant material properties database, now approaching 50 years of maturity, is valuable not only because these materials are still being used, but also because it serves as a foundation for predicting how other materials will perform under similar conditions—and in the space programs of the future.

Space Environment: It's More Than a Vacuum

We know that materials behave differently in different environments on Earth. For example, aluminum does not change on a pantry shelf for years yet rapidly corrodes or degrades in salt water.

One would think that such material degradation effects would be eliminated by going to the near-perfect vacuum of space in low-Earth orbit. In fact, many of these effects are eliminated. However, Orbiter systems produced gas, particles, and light when engines, overboard dumps, and other systems operated, thereby creating an induced environment in the immediate vicinity of the spacecraft. In addition, movement of the shuttle through the tenuous upper reaches of Earth's atmosphere (low-Earth orbit) at orbital velocity produced additional contributions to the induced environment in the form of spacecraft glow and atomic oxygen effects on certain materials. The interactions of spacecraft materials with space environment factors like solar ultraviolet (UV) light, atomic oxygen, ionizing radiation, and extremes of temperature can actually be detrimental to the life of materials used in spacecraft systems.

For the Orbiter to perform certain functions and serve as a platform for scientific measurements, the effects of natural and Orbiter-induced environments had to be evaluated and controlled. Payload sensitivities to these environmental effects varied, depending on payload characteristics. Earth-based observatories and other instruments are affected by the Earth's atmosphere in terms of producing unwanted light background and other contamination effects. Therefore, NASA developed



essential analytical tools for environment prediction as well as measurement systems for environment definition and performance verification, thus enabling a greater understanding of natural and induced environment effects for space exploration.

Induced Environment Characterization

NASA developed mathematical models to assess and predict the induced environment in the Orbiter cargo bay during the design and development phase of the Space Shuttle Program. Models contained the vehicle geometry, vehicle flight attitude, gas and vapor emission source characteristics, and used low-pressure gas transport physics to calculate local gas densities, column densities (number of molecular species seen along a line of sight), as well as contaminant deposition effects on functional surfaces. Gas transport calculations were based on low-pressure molecular flow physics and included scattering from Orbiter surfaces and the natural low-Earth orbit environment.

The Induced Environment Contamination Monitor measured the induced environment on three missions—Space Transportation System (STS)-2 (1981), STS-3 (1982), and STS-4 (1982)—and was capable of being moved using the Shuttle Robotic Arm to various locations for specific measurements. Most measurements were made during the on-orbit phase. This measurement package was flown on the three missions to assess shuttle system performance. Instruments included a humidity monitor, an air sampler for gas collection and analysis after return, a cascade impactor for particulate measurement, passive samples for optical degradation of



The Atlantic Ocean southeast of the Bahamas is in the background as Columbia's Shuttle Robotic Arm and end effector grasp a multi-instrument monitor for detecting contaminants. The experiment, called the Induced Environment Contaminant Monitor, was flown on STS-4 (1982). The tail of the Orbiter can be seen below.

surfaces, quartz-crystal microbalances for deposited mass measurement, a camera/photometer pair for particle measurement in the field of view, and a mass spectrometer. Additional flight measurements made on STS-52 (1992) and many payloads provided more data.

Before the induced environment measurements could be properly interpreted, several on-orbit operational aspects needed to be understood. Because of the size of the vehicle and its payloads, desorption of adsorbed gases such as water, oxygen, and nitrogen (adsorbed on Earth) took a fairly long time, the induced environment on the first day of a mission was affected more than on

subsequent days. Shuttle flight attitude requirements could affect the cargo bay gaseous environment via solar heating effects as well as the gases produced by engine firings. These gases could reach the payload bay by direct or scattered flow. Frequently, specific payload or shuttle system attitude or thermal control requirements conflicted with the quiescent induced environment required by some payloads.

With the above operational characteristics, data collected with the monitor and subsequent shuttle operations showed that, in general, the measured data either met or were close to the requirements of sensitive payloads during quiescent periods. A large qualification to this statement



had to be made based on a new understanding of the interaction of the natural environment with vehicle surfaces. This interaction resulted in significantly more light emissions and material surface effects than originally expected. Data also identified an additional problem of recontact of particles released from the shuttle during water dumps with surfaces in the payload bay. The induced environment control program instituted for the Space Shuttle Program marked a giant step from the control of small free-flying instrument packages to the control of a large and complex space vehicle with a mixed complement of payloads. This approach helped develop a system with good performance, defined the vehicle associated environment, and facilitated effective communication between the program and users.

The induced environment program also showed that some attached payloads were not compatible with the shuttle system and its associated

payloads because of the release of water over long periods of time. Other contamination-sensitive payloads such as Hubble Space Telescope, however, were not only successfully delivered to space but were also repaired in the payload bay.

Unique Features Made It Possible

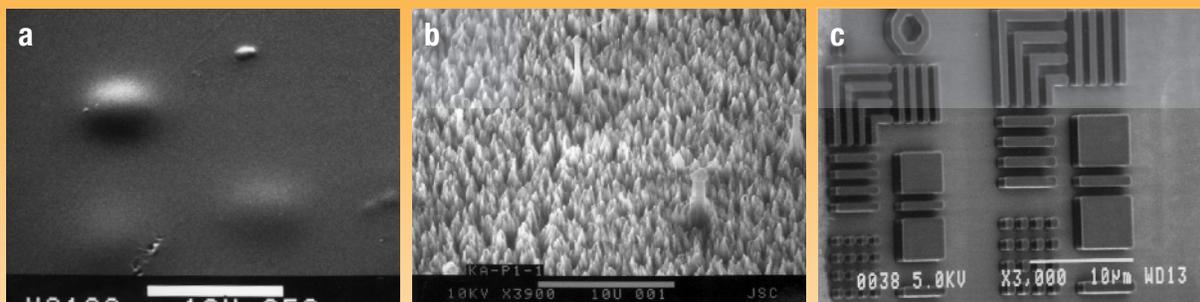
The Orbiter was the first crewed vehicle to provide protection of instrumentation and sensitive surfaces in the payload bay during ascent and re-entry and allow exposure to the low-Earth orbit environment. Effects were observed without being modified by flight heating or gross contamination. Also, as part of the induced environment control program, the entire payload bay was examined immediately on return. Because of these unique aspects, NASA was able to discover and quantify unexpected interactions between the environment of low-Earth and the vehicle.

Discovery of Effects of Oxygen Atoms

After STS-1 (1981) returned to Earth, researchers visually examined the material surfaces in the payload bay for signs of contamination effects. Most surfaces appeared pristine, except for the exterior of the television camera thermal blankets and some painted surfaces. The outside surface of the blankets consisted of an organic (polyimide) film that, before flight, appeared gold colored and had a glossy finish. After flight, most films were altered to a yellow color and no longer had a glossy finish but, rather, appeared carpet-like under high magnification. Only the surfaces of organic materials were affected; bulk properties remained unchanged.

Patterns on modified surfaces indicated directional effects and, surprisingly, the flight-exposed surfaces were found to have receded rather than having deposited contaminants. The patterns on the surfaces were related to the

Atomic Oxygen Effects on Polymers and Plastics in low-Earth Orbit as Seen With the Scanning Electron Microscope; STS-46 (1992)



- Scanning electron microscope image of a typical Kapton® polyimide plastic sheet. The various specs and bumps are from the inorganic filler used in plastic sheet manufacture.
- Scanning electron microscope image of a typical Kapton® polyimide plastic sheet after exposure to surface bombardment by atomic oxygen in low-Earth orbit. The rough surface is typical of atomic oxygen attack on plastics in low-Earth orbit and is the result of the strong dependence of chemical reaction on atom-surface collision energy. Note how some of the inorganic filler particles are standing on pedestals because they protect the underlying plastic from atomic oxygen attack.
- Scanning electron microscope image of a microelectron fabrication etching target also flown on STS-46 and exposed to low-Earth orbit atomic oxygen. The highly directional attack of low-Earth orbit atomic oxygen produced a clean, high-resolution removal of the unprotected plastic around the pattern of protective inorganic surface coatings. High-speed neutral atomic oxygen beams in ground-based production facilities may be a useful adjunct to microelectronic production as described in US Patent 5,271,800.



vehicle velocity vector. When combining these data with the atmospheric composition and densities, the material surface recession was caused by the high-velocity collision of oxygen atoms with forward-facing Orbiter surfaces leading to surface degradation by oxidation reactions. Oxygen atoms are a major constituent of the natural low-Earth orbit environment through which the shuttle flew at an orbital velocity of nearly 8 km/sec (17,895 mph). The collision energy of oxygen atoms striking forward-facing shuttle surfaces in low-Earth orbit was extremely high—on the order of 5 electron volts (eV)—100 times greater than the energy of atoms in typical low-pressure laboratory oxygen atom generators. The high collision energy of oxygen atoms in low-Earth orbit plays an important role in surface reactivity and surface recession rates.

Material recession rates are determined by normalizing the change in sample mass to the number of oxygen atoms reaching the surface over the exposure time (atoms/cm², fluence). Atom density is obtained from the standard atmospheric density models used by NASA and the Department of Defense. Since oxygen atoms travel much slower than the Orbiter, they impacted the surfaces in question only when facing toward the vehicle velocity vector and had to be integrated over time and vehicle orientation. STS-1 recession data were approximate because they had to be integrated over changing vehicle attitude; had limited atom flux, uncontrolled surface temperatures and solar UV exposure; and predicted atom densities. Recession rates determined from material samples exposed during the STS-5 (1982) mission and Induced Environmental Contamination Monitor

flights had the same limitations but supported the STS-1 data. Extrapolation of these preliminary recession data to longer-term missions showed the potential for significant performance degradation of critical hardware, so specific flight experiments were carried out to quantify the recession characteristics and rates for materials of interest.

On-orbit Materials Behavior

Fifteen organizations participated in a flight experiment on STS-8 (1983) to understand materials behavior in the low-Earth orbit environment. The objective was to control some of the parameters to obtain more-accurate recession rates. The mission had a dedicated exposure to direct atom impact (payload bay pointing in the velocity direction) of 41.7 hours at an altitude of 225 km (121 nautical miles) resulting in the largest fluence of the early missions (3.5×10^{20} atoms/cm²). Temperature control at two set points was provided as well as instruments to control UV and exposure to electrically charged ionospheric plasma species.

The STS-8 experiment provided significant insight into low-Earth orbit environment interactions with materials. Researchers established quantitative reaction rates for more than 50 materials, and were in the range of $2\text{--}3 \times 10^{-24}$ cm³/atom for hydrocarbon-based materials. Perfluorinated organic materials were basically nonreactive and silicone-based materials stopped reacting after formation of a protective silicon oxide surface coating. Material reaction rates, as a first approximation, were found to be independent of temperature, material morphology, and exposure to solar radiation or electrically charged ionospheric species.

Researchers also evaluated coatings that could be used to protect surfaces from interaction with the environment.

Reaction rates were based on atomic oxygen densities determined from long-term atmospheric density models, potentially introducing errors in short-term experiment data. In addition, researchers obtained very little insight into the reaction mechanism(s).

An additional flight experiment—Evaluation of Oxygen Interaction with Materials III—addressing both of these questions was flown on STS-46 (1992). The primary objective was to produce benchmark atomic oxygen reactivity data by measuring the atom flux during material surface exposure. Secondary experiment objectives included: characterizing the induced environment near several surfaces; acquiring basic chemistry data related to reaction mechanism; determining the effects of temperature, mechanical stress, atom fluence, and solar UV radiation on material reactivity; and characterizing the induced and contamination environments in the shuttle payload bay. This experiment was a team effort involving NASA centers, US Air Force, NASA Space Station Freedom team, Aerospace Corporation, University of Alabama in Huntsville, National Space Agency of Japan, European Space Agency, and the Canadian Space Agency.

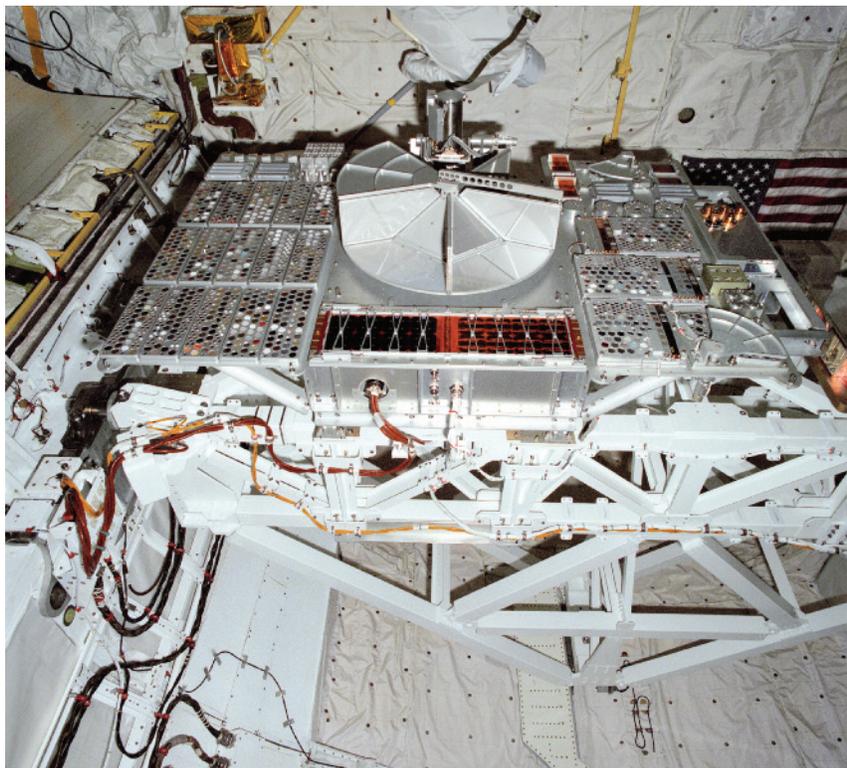
STS-46 provided an opportunity to make density measurements at several altitudes: 427, 296, and 230 km (231, 160, and 124 nautical miles). However, the vehicle flew for 42 hours at 230 km (124 nautical miles) with the payload bay surfaces pointed into the velocity vector during the main portion of the mission to obtain high fluence. The mass spectrometer provided by the



US Air Force was the key component of the experiment and was capable of sampling both the direct atomic oxygen flux as well as the local neutral environment created by interaction of atomic oxygen with surfaces placed in a carousel. Five carousel sections were each coated with a different material to determine the material effects on released gases. Material samples trays, which provided temperature control plus instruments to control other exposure conditions, were placed on each side of the mass spectrometer/carousel.

NASA achieved all of the Evaluation of Oxygen Interaction with Materials III objectives during STS-46. A well-characterized, short-term, high-fluence atomic oxygen exposure was provided for a large number of materials, many of which had never been exposed to a known low-Earth orbit atomic oxygen environment. The data provided a benchmark reaction rate database, which has been used by the International Space Station, Hubble, and others to select materials and coatings to ensure long-term durability.

Reaction rate data for many of the materials from earlier experiments were confirmed, as was the generally weak dependence of these reaction rates on temperature, solar UV exposure, oxygen atom flux, and exposure to charged ionospheric species. The role of surface collision energy on oxygen atom reactivity was quantified by comparing flight reaction rates of key Evaluation of Oxygen Interaction with Materials III experiment materials with reactivity measurements made in well-characterized laboratory oxygen atom systems with lower surface collision energies. This evaluation also provided an important benchmark point for understanding the role of



Evaluation of Oxygen Interaction with Materials III flight experiment in the Orbiter payload bay of STS-46 (1992). Material exposure samples are located on both sides of the mass spectrometer gas evolution measurement assembly in the center.

solar extreme UV radiation damage in increasing the generally low surface reactivity of perfluorinated organic materials. The mass spectrometer/carousel experiment produced over 46,000 mass spectra providing detailed characterization of both the natural and the induced environment. The mass spectrometer database provided a valuable resource for the verification of various models of rarified gas and ionospheric plasma flow around spacecraft.

Intelsat Satellite

Knowledge gained from atomic oxygen reactivity studies played a key role in the STS-49 (1992) rescue of the communications satellite

Intelsat 603 that was used to maintain communications from a geosynchronous orbit. Failure of the Titan-3 upper stage left Intelsat 603 marooned in an unacceptable low-Earth orbit and subject to the effects of atomic oxygen degradation of its solar panels, which could have rendered the satellite useless. NASA quickly advised the International Telecommunications Satellite Organization (Intelsat) Consortium of the atomic oxygen risk to Intelsat 603, leading to the decision to place the satellite in a configuration that was expected to minimize atomic oxygen damage to the silver interconnects on the solar panels. This was accomplished by raising the satellite altitude and changing its flight attitude so that atomic oxygen fluence was minimized.



The Intelsat Solar Array Coupon flight experiment shown mounted on the Shuttle Robotic Arm lower arm boom and exposed to space environment conditions during STS-41 (1990).

To provide facts needed for a final decision about a rescue flight, NASA designed and executed the Intelsat Solar Array Coupon flight experiment on STS-41 (1990). The experiment results, in combination with ground-based testing, supported the decision to conduct the STS-49 satellite rescue mission. On this mission, Intelsat 603 was captured and equipped with a solid re-boost motor to carry it to successful geosynchronous orbit.

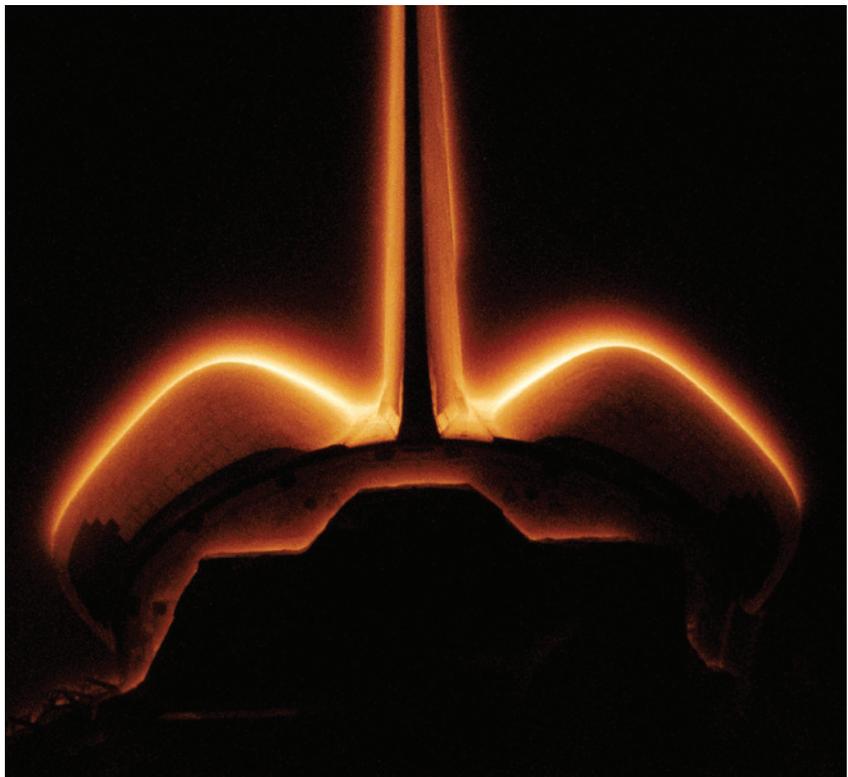
STS-62 (1994) orbits Earth during a “night” pass, documenting the glow phenomenon surrounding the vertical stabilizer and the Orbital Maneuvering System pods of the spacecraft.

NASA Discovers Light Emissions

On the early shuttle flights, NASA observed another effect caused by the interaction between spacecraft surfaces and the low-Earth orbit environment. Photographs obtained by using intensified cameras and conducted from the Orbiter cabin windows showed light emissions (glow) from the Orbiter surfaces when in forward-facing conditions.

The shuttle provided an excellent opportunity to further study this phenomenon. On STS-41D (1984), astronauts photographed various material samples using a special glow spectrometer to obtain additional data and determine if the glow was dependent on surface composition. These measurements, along with the material recession effects and data obtained on subsequent flights, led to a definition of the glow mechanism.

Spacecraft glow is caused by the interaction of high-velocity oxygen atoms with nitrous oxide absorbed on the surfaces, which produces nitrogen dioxide in an electronically excited state. The excited nitrogen dioxide is released from the surfaces and emits light as it moves away and decays from its excited state. Some nitrous oxide on the surface and some of the released nitrogen dioxide result from the natural environment. The light emission occurs on any spacecraft operating in low-Earth orbit; however, the glow could be enhanced by operation of the shuttle attitude control engines, which produced nitrous oxide and nitrogen dioxide as reaction products. These findings led to a better understanding of the behavior of spacecraft operating in low-Earth orbit and improved accuracy of instrument measurements.





Chemical Fingerprinting

Comprehensive Electronic System for Greater Flight Safety

A critical concern for all complex manufacturing operations is that contaminants and material changes over time can creep into the production environment and threaten product quality. This was the challenge for the solid rocket motors, which were in production for 30 years.

It is possible that vendor-supplied raw materials appear to meet specifications

from lot to lot and that supplier process changes or even contaminated material can appear to be “in spec” but actually contain subtle, critical differences. This situation has the potential to cause significant problems with hardware performance.

NASA needed a system to readily detect those subtle yet potentially detrimental material variances to ensure the predictability of material properties and the reliability of shuttle reusable solid rocket motors. The envisioned solution was to pioneer consistent and repeatable analytical methods tailored to specific, critical materials that would yield accurate assessments of material

integrity over time. Central to the solution was both a foolproof analysis process and an electronic data repository for benchmarking and monitoring.

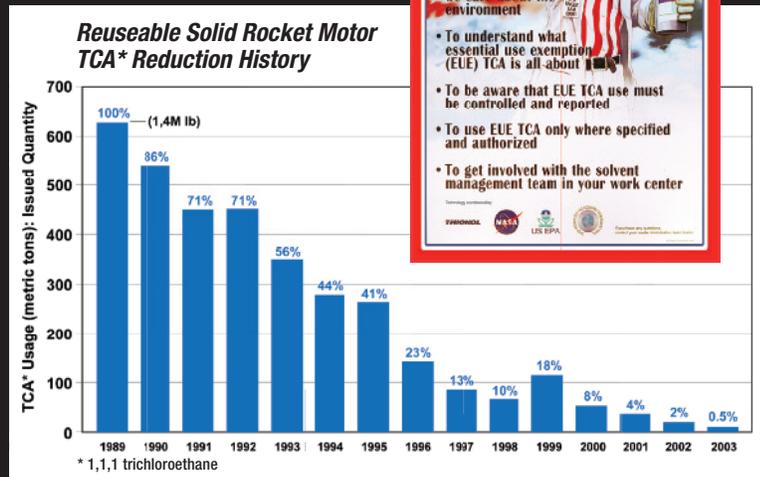
A Chemical “Fingerprint”

Just as fingerprints are a precise method to confirm an individual’s identity, the solid rocket motor project employed chemical “fingerprints” to verify the quality of an incoming raw material. These fingerprints comprised a detailed spectrum of a given material’s chemical signature, which could be captured digitally and verified using a combination of sophisticated laboratory equipment and custom analytical methods.

The challenge was to accurately establish a baseline chemical fingerprint of each material and develop reproducible analytical test methods to monitor lot-to-lot material variability. A further objective was to gain a greater understanding of critical reusable solid rocket motor materials, such as insulation and liner ingredients, many of which were the same materials used since the Space Shuttle Program’s inception. New analytical techniques such as the atomic force microscope were used to assess materials at fundamental chemical, molecular, and mechanical levels. These new techniques provided the high level of detail sought. Because of unique attributes inherent in each material, a one-size-fits-all analysis method was not feasible.

To facilitate documentation and data sharing, the project team envisioned a comprehensive electronic database to provide ready access to all relevant data. The targeted level of background detail included everything from where and how a material was properly used to details of chemical composition.

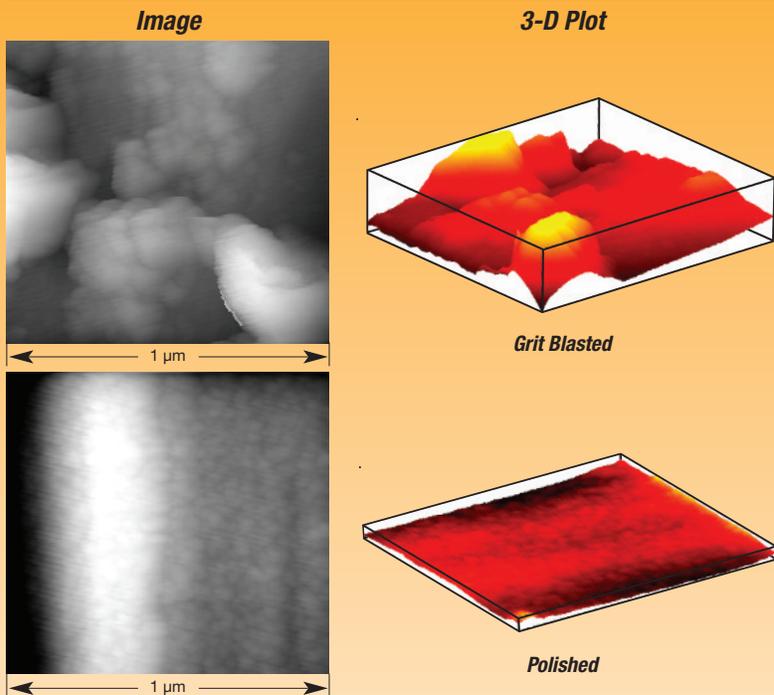
Environmental Assurance



During the Space Shuttle Program’s operation, issues arose regarding the use of substances that did not meet emerging environmental regulations and current industry standards. NASA worked to develop chemicals, technologies, and processes that met regulatory requirements, and the agency strove to identify, qualify, and replace materials that were becoming obsolete as a result of environmental issues. The stringent demands of human spaceflight required extensive testing and qualification of these replacement materials.



Tools for Materials Evaluation
Atomic Force Microscope Images of Metal Surface



The atomic force microscope affords a visual evaluation of surface preparation processes to improve understanding of their effects on bonding. The top panel represents topography of a grit blast surface for comparison to a highly polished one. The atomic force microscope uses an extremely fine probe to measure minute interactions with surface features even down to an atomic scale. The maps at left are scaled from black at the bottom of valleys to white at the tops of peaks within the scanned area. The 3-D projections at right are on a common height scale. The grit blast surface clearly offers greatly increased surface area and mechanical interlocking for enhanced bonding. Beyond simple topography, the probe interactions with atomic forces can also measure and map properties such as microscopic hardness or elastic modulus on various particles and/or phase transitions in a composite material, which in turn can be correlated with chemical and physical properties.

© ATK. All rights reserved.

The ideal system would enable a qualified chemist to immediately examine original chemical analysis data for the subtle yet significant differences between the latest lot of material and previous good or bad samples.

To develop such a system, commercially available hardware and software were used to the greatest extent possible. Since an electronic framework to tie the data together did not exist, one was designed in-house.

The Fingerprinting Process

The chemical fingerprinting program, which began in 1998 with a prioritized list of 14 critical materials, employed a team approach to quantify and document each material. The interdisciplinary team included design engineering, materials and processes engineering, procurement quality engineering, and analytical chemistry. Each discipline group proposed test plans that included the types of testing to be developed. Following approval,

researchers acquired test samples (usually three to five lots of materials) and developed reliable test methods. Because of the unique nature of each material, test methods were tailored to each of the 14 materials.

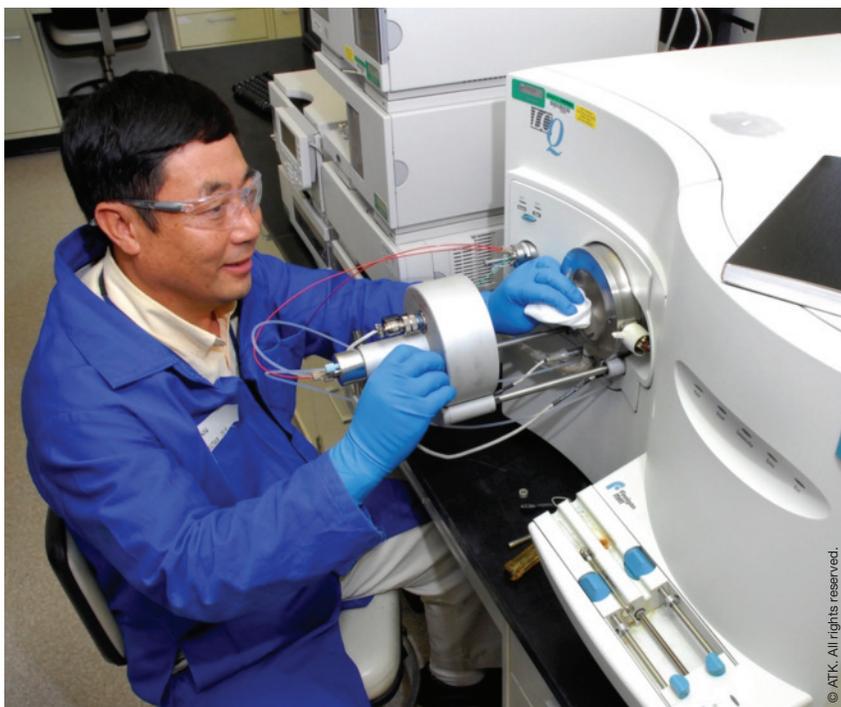
A “material” site in the project database was designed to ensure all data were properly logged and critical reports were written and filed. Once the team agreed sufficient data had been generated, a formal report was drafted and test methods were selected to develop new standard acceptance procedures that would ultimately be used by quality control technicians to certify vendor materials.

The framework developed to package the wide-ranging data was termed the Fingerprinting Viewer. Program data were presented through a series of cascading menu pages, each with increasing levels of detail.

The Outcomes

Beyond meeting the primary program objectives, a number of resulting benefits were noted. First, through increased data sharing, employees communicated more effectively, both internally and with subtier suppliers. The powerful analytical methods employed also added to the suppliers’ materials knowledge base. Subtle materials changes that possibly resulted from process drift or changes at subtier suppliers were detectable. Eight subtier suppliers subsequently implemented their own in-house chemical fingerprinting programs to improve product consistency, recertify material after production changes, or even help develop key steps in the manufacturing process to ensure repeatable quality levels.

Additionally, engineers could now accurately establish shelf-life extensions and storage requirements



This high-performance liquid chromatography/mass spectrometry is employed to document minute details of a material's chemical and molecular composition. Through the chemical fingerprinting system, seemingly minuscule discrepancies raise red flags that trigger investigations and preclude defective materials from reaching the production floor. Dr. Ping Li shown here at ATK in Utah.

for stockpiled materials. The ability to store greater amounts of materials over longer periods of time was valuable in cases where new materials needed to be certified to replace existing materials that had become obsolete.

Finally, investigators were able to solve production issues with greater efficiency. Comprehensive database features, including standardized test methods and the extensive online reference database, provided resources needed to resolve production issues in a matter of days or even hours—issues that otherwise would have required major investigations. In some cases, fingerprinting was also used to indicate that a suspect material was actually within required specifications. These materials may have been rejected in previous cases but, by using the fingerprinting database to assess the

material, the team could look deeper to find the true root cause and implement proper corrective actions.

From Fingerprints to Flight Safety

The overarching value of the chemical fingerprinting program was that it provided greater assurance of the safety and reliability of critical shuttle flight hardware. The fundamental understanding of critical reusable solid rocket motor materials and improved communications with vendors reduced the occurrence of raw materials issues. NASA will implement chemical fingerprinting methods into the acceptance testing of raw materials used in future human space exploration endeavors. The full benefits of the program will continue to be realized in years to come.

Unprecedented Accomplishments in the Use of Aluminum-Lithium Alloy

NASA was the first to use welded aluminum-lithium alloy Al 2195 at cryogenic temperatures, incorporating it into the External Tank under circumstances that demanded innovation.

From the beginning of the Space Shuttle Program's launch phase, NASA sought to reduce the weight of the original tank, thereby increasing payload capacity. Since the tank was carried nearly to orbit, close to 100% of the weight trimmed could be applied to the payload. NASA succeeded in implementing numerous weight-saving measures, but the biggest challenge was to incorporate a lightweight aluminum alloy—aluminum-lithium Al 2195—into the tank structure. This alloy had never been used in welded cryogenic environments prior to NASA's initiative. Several challenges needed to be overcome, including manufacturing the aluminum-lithium tank components, welding the alloy, and repairing the welds. NASA and the External Tank prime contractor broke new ground in the use of aluminum-lithium to produce the "super lightweight tank."

The original tank weighed 34.500 metric tons (76,000 pounds) dry. By the sixth shuttle mission, the tank's weight had been reduced to 29.900 metric tons (66,000 pounds). This configuration was referred to as the "lightweight tank."

The real challenge, however, was still to come. In 1993, the International Space Station Program decided to change the station's orbital inclination



to 57 degrees (a “steeper” launch inclination), allowing Russian vehicles to fly directly to the station. That change cost the shuttle 6,123 kg (13,500 pounds) of payload capacity. The External Tank project office proposed to reduce the dry weight of the tank by 3,402 kg (7,500 pounds).

The Space Shuttle Program sought to incorporate lightweight aluminum-lithium Al 2195 into the majority of the tank structure, replacing the original aluminum-copper alloy Al 2219; however, NASA first needed to establish requirements for manufacturing, welding, and repairing aluminum-lithium weld defects.

NASA started the super lightweight tank program in 1994. During the early phase, advice was sought from welding experts throughout the United States and the United Kingdom. The consensus: it was virtually impossible to perform repairs on welded aluminum-lithium.

The aluminum-lithium base metal also presented challenges. Lockheed Martin worked with Reynolds Aluminum to produce the aluminum-lithium base metal. One early problem was related to aluminum-lithium material’s fracture toughness—a measure of the ability of material with a defect to carry loads. Although material was screened, flight hardware requirements dictated that structures must have the ability to function in the event a defect was missed by the screening process. The specific difficulty with the aluminum-lithium was that the cryogenic fracture toughness of the material showed little improvement over the room-temperature fracture toughness.

Since the two propellant tanks were proof tested at room temperature and flown cryogenically, this fracture toughness ratio was a crucial factor.

A simulated service test requirement was imposed as part of lot acceptance for all aluminum-lithium material used on the tank. The test consisted of applying room temperature and cryogenic load cycles to a cracked sample to evaluate the ability of the material to meet the fracture toughness requirements. Failure resulted in the plate being remelted and reprocessed.

Implementation of simulated service testing as a lot acceptance requirement was unique to the aluminum-lithium material. Testing consisted of cropping two specimens from the end of each plate. Electrical discharge machining (a process that removes metal by discharging a spark between the tool and the test sample) was used to introduce a fine groove in each sample. The samples were then cyclically loaded at low stresses to generate a sharp fatigue crack that simulated a defect in the material.

The first sample was stressed to failure; the second sample was stressed to near failure and then subjected to cyclic loading representative of load cycles the tank would see on the launch pad during tanking and during flight.

In the second sample, initial loading was conducted at room temperature. This simulated the proof test done on the tank. Next, the sample was stressed 13 times (maximum tanking requirement) to the level expected during loading of propellants at cryogenic temperatures and, finally, stressed to maximum expected flight

stress at cryogenic temperature. This cycle was repeated three more times to meet a four-mission-life program requirement with the exception that, on the fourth cycle, the sample was stressed to failure and had to exceed a predetermined percent of the flight stress. Given the size of the barrel plates for the liquid hydrogen and liquid oxygen tanks, only one barrel plate could be made from each lot of material. As a result, this process was adopted for every tank barrel plate—32 in each liquid hydrogen tank and four in each liquid oxygen tank—and implemented for the life of the program.

Another challenge was related to the aluminum-lithium weld repair process on compound curvature parts. The effect of weld shrinkage in the repairs caused a flat spot, or even a reverse curvature, in the vicinity of the repairs and contributed to significant levels of residual stress in the repair. Multiple weld repairs, in proximity, showed the propensity for severe cracking. After examination of the repaired area, it was found that welding aluminum-lithium resulted in a zone of brittle material surrounding the weld. Repeated repairs caused this zone to grow until the residual stress from the weld shrinkage exceeded the strength of the weld repair, causing it to crack.

The technique developed to repair these cracks was awarded a US Patent. The repair approach consisted of alternating front-side and back-side grinds as needed to remove damaged microstructure. It was also found that aluminum-lithium could not tolerate as much heating as the previous aluminum-copper alloy. This required increased torch speeds and decreased



The use of aluminum-lithium Al 2195 in manufacturing major External Tank components, such as the liquid hydrogen tank structure shown above, allowed NASA to reduce the overall weight of the External Tank by 3,402 kg (7,500 pounds). The liquid hydrogen tank measured 8.4 m (27.5 ft) in diameter and 29.4 m (96.6 ft) in length. Photo taken at NASA's Michoud Assembly Facility in New Orleans, Louisiana.

fill volumes to limit the heat to which the aluminum-lithium was subjected.

Additional challenges in implementing effective weld repairs caused NASA to reevaluate the criteria for measuring the strength of the welds. In general, weld repair strengths can be evaluated by excising a section of the repaired material and performing a tensile test. The strength behavior of the repaired material is compared to the strength behavior of the original weld material. In the case of the aluminum-copper alloy Al 2219, the strengths were

comparable; however, in the case of the aluminum-lithium alloy repair, the strengths were lower.

Past experience and conventional thinking was that in the real hardware, where the repair is embedded in a long initial weld, the repaired weld will yield and the load will be redistributed to the original weld, resulting in higher capability. To demonstrate this assumption, a tensile test was conducted on a 43-cm- (17-in.-) wide aluminum-lithium panel that was fabricated by welding two

aluminum-lithium panels together and simulating a weld repair in the center of the original weld. The panel was then loaded to failure. The test that was supposed to indicate better strength behavior than the excised repair material actually failed at a lower stress level.

To understand this condition, an extensive test program was initiated to evaluate the behavior of repairs on a number of aluminum-copper alloy (Al 2219) and aluminum-lithium alloy (Al 2195) panels.

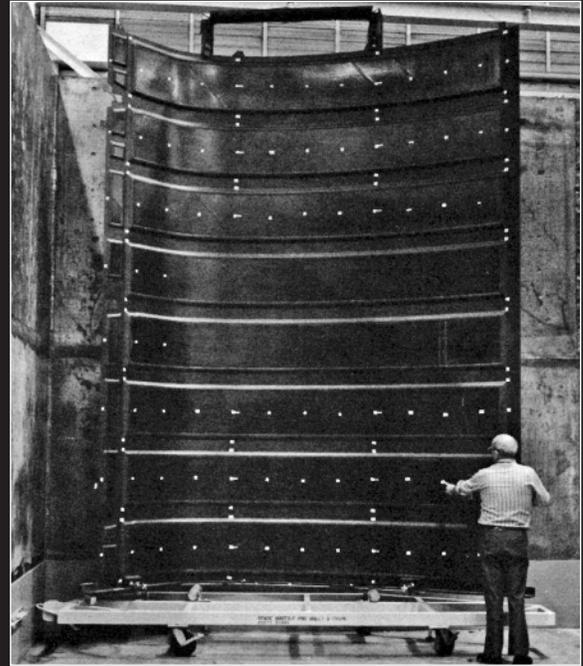


Orbiter Payload Bay Door

One of the largest aerospace composite applications of its time.

With any space vehicle, minimum weight is of critical importance. Initial trade studies indicated that using a graphite/epoxy structure in place of the baselined aluminum structure provided significant weight savings of about 408 kg (900 pounds [4,000 newtons]), given the large size and excellent thermal-structural stability. Two graphite/epoxy composite materials and four structural concepts—full-depth honeycomb sandwich, frame-stiffened thin sandwich, stiffened skin with frames and stringers, and stiffened skin with frames only—were considered for weight savings and manufacturing producibility efficiency. These studies resulted in the selection of the frame-stiffened thin sandwich configuration, and component tests of small specimens finalized the graphite fiber layup, matrix material, and honeycomb materials. Graphite/epoxy properties at elevated temperatures are dependent on moisture content and were taken into account in developing mechanical property design allowables. Additionally, NASA tracked the moisture content through all phases of flight to predict the appropriate properties during re-entry when the payload bay doors encountered maximum temperatures of 177°C (350°F).

Payload bay doors were manufactured in 4.57-m (15-ft) sections, resulting in two 3 x 18.3 m (10 x 60 ft) doors. The panel face sheets consisted of a ± 45-degree fabric ply imbedded between two 0-degree tape plies directed normal to the frames and were pre-cured prior to bonding to the Nomex® honeycomb core. A lightweight-aluminum wire mesh bonded to the outside of face sheets provided lightning-strike protection. Frames consisted primarily of fabric plies with the interspersions of 0-degree plies dictated by strength and/or



stiffness. Mechanical fasteners were used for connection of major subassemblies as well as final assembly of the doors.

All five Orbiter vehicles used graphite/epoxy doors, one of the largest aerospace composite applications at the time, and performance was excellent throughout all flights. Not only was the expected weight saving achieved and thermal-structural stability was acceptable, NASA later discovered that the graphite/epoxy material showed an advantage in ease of repair. Ground handling damage occurred on one section of a door, resulting in penetration of the outer skin of the honeycomb core. The door damage was repaired in 2 weeks, thereby avoiding significant schedule delay.



Test panels were covered with a photo-stress coating that, under polarized light, revealed the strain pattern in the weld repair. The Al 2219 panel behaved as expected: the repair yielded, the loads redistributed, and the panel pulled well over the minimum allowable value. In aluminum-lithium panels, however, the strains remained concentrated in the repair. Instead of the 221 MPa (32,000 pounds/in²) failure stress obtained in the initial welds, the welds were failing around 172 MPa (18,000 pounds/in²). These lower failure stress values were problematic due to a number of flight parts that had already been sized and machined for the higher 221 MPa (32,000 pounds/in²) value.

Based on this testing, it was determined that weld shrinkage associated with the repair resulted in residual stresses in the joint, reducing the joint capability. To improve weld repair strengths, engineers developed an approach to planish (lightly hammer) the weld bead, forcing it back into the joint and spreading the joint to redistribute and reduce the residual stresses due to shrinkage. This required scribing and measuring the joint before every repair, making the repair, and then planishing the bead to restore the weld to its previous dimensions. Wide panel test results and photo-stress evaluation of planished repairs revealed that the newly devised repair procedure was effective at restoring repair strengths to acceptable levels.

Testing also revealed that planishing of weld beads is hard to control precisely, resulting in the process frequently forming other cracks, thus leading to additional weld repairs. Because of the

difficulty in making and planishing multiple repairs, a verification ground rule was established that every “first repair of its kind” had to be replicated on three wide tensile panels, which were then tested either at room temperature or in a cryogenic environment, depending on the in-flight service condition expected for that part of the tank.

All these measures combined accomplished the first-ever use of welded aluminum-lithium at cryogenic temperatures, meeting the strict demands of human spaceflight. The super lightweight tank incorporated 20 aluminum-lithium ogive gores (the curved surfaces at the forward end of the liquid oxygen tank), four liquid oxygen barrel panels, 32 liquid hydrogen barrel panels, 12 liquid oxygen tank aft dome gores, 12 liquid hydrogen tank forward dome gores, and 11 liquid hydrogen aft dome gores.

Through this complex and innovative program, NASA reduced the 29,937-kg (66,000-pound) lightweight tank by another 3,401.9 kg (7,500 pounds). The 26,560-kg (58,500-pound) super lightweight tank was first flown on Space Transportation System (STS)-91 (1998), opening the door for the shuttle to deliver the heavier components needed for construction of the International Space Station.